

**FunHy Meeting: Institutt for Energi (IFE), 17.-18. December 2018.**

## Minutes from the meeting

Short link to our homepage: <http://inano.au.dk/FunHy>

Share point: <https://aarhusuniversitet.sharepoint.com/sites/funhy-Nordforsk>

Login details: An AU-login might be necessary to access the sharepoint.

**Participants:** Martin Sahlberg, Gustav Ek, Magnus H. Sørby, Magnus Moe Nygård, Bjørn C. Hauback, Maths Karlsson, Mikael Svante Andersson, Torben Jensen, Jakob B. Grinderslev

**Meeting room:** in IFE's chemistry building

**Local organisers:** Bjørn Hauback (mobile +47 974 08 844), Magnus H. Sørby (mobile +47 900 82 208) and Magnus Moe Nygård (mobile +47 414 26 626)

**Accommodation:** Thon Hotel Lillestrøm (reservation number 337831255)

### Monday 17. December

12-13 Arrival and Lunch

13:00-13.10 Welcome by Bjørn and Magnus.

13:10-14.30 Torben, FunHy status and yearly report for 2018 (to be submitted, early 2019).  
General discussion of FunHy progress. Reminder about next reporting to NordForsk and what we promised in the original proposal

Continued discussions of how to organise 'open data' exchange of samples and data, update of home page. Initial discussion of next beam time proposals and beam times (develop a plan to be further discussed). Final discussion of these topics Tuesday.

Update from responsible **PhD students:**

- **Webmaster** Jakob, input to web page, FunHy-web with scientific profile, activities: Jakob call for more contributions to the homepage. He suggest to make a short (1-page) news of published papers, e.g. the title, authors, abstract and 1-2 significant figures.
- **Data management plan**, Gustav. Intranet sharing data and results, FTP-server site for data sharing. The share point at the FunHy web-page should be used more. We work on that.
- **Beamtime allocations**, plan for beamtime proposals, participants, research topic, Magnus will make a shared google spreadsheet)  
Petra beamline has proposal deadline marts 2019. Martin suggest common FunHy proposal. One may be on high pressure hydrogen reactions.  
Link to spreadsheet:  
<https://docs.google.com/spreadsheets/d/1vK7kAXvi6Hn3J90b7BxDp3V8btJ-Z1i155XGu1ULNY4/edit#gid=0>

- **Dissemination to the public**, Magnus. Newsletter, Public summary, activities and results, popular science. We agree to focus on public news, shortly describing published papers by FunHy PhDs

14:30 Coffee.

14:45-15:15 **“Funhy and neutrons at NIST” by Mikael Svante Andersson**

Mikael has since June been located at the NIST Center for Neutron Research (NCNR) in Maryland, USA, and he will stay there until June 2020. At NCNR he has good access to beam time He gave a short presentation about NIST and the NCNR facility, as well as a brief overview of the QENS technique. Mikael also went through which systems that have been studied since the last meeting (April) and the status of the corresponding projects:

- $\text{NH}_4\text{BH}_4$ . Two manuscripts in preparation, one on the QENS results, which Mikael is writing and one on the INS results, which Ulrich is writing.
- $\text{Y}(\text{BH}_4)_3\text{-xNH}_3$ . Further samples are needed to complete the study, Jakob will provide the last samples in January/February
- $\text{Pr}(\text{BH}_4)_3\text{-xNH}_3$ . Analysis is currently ongoing; the results will probably be combined with the  $\text{Y}(\text{BH}_4)_3\text{-xNH}_3$ . It is currently not certain if the results will be presented in one INS and one QENS article, or as a larger article containing all of the results.
- $\text{KB}_3\text{H}_8$ . Used partly as a test sample to see if measurements could be made on samples containing natural boron. There are some initial results however more beam time is needed to complete the study.

15:15-16:30 PhD-research project presentation (by Gustav, Magnus and Jakob, 30 min each) and discussions. Planning of new collaborative research activities. (first part)

**Jakob B. Grinderslev:** Jakob will have a research stay in Norway (IFE) from February-April, and a research stay in Sweden (Uppsala University) in May. Jakob gave a presentation on rare earth metal borohydrides and ammonium metal borohydrides. Projects in relation to the samples investigated by Mikael at ISIS and NIST were briefly discussed.

- $\text{RE}(\text{BH}_4)_x$ : A manuscript has been submitted based on the synthesis, crystal structures, thermal and magnetic properties of the complete series of halide-free rare earth (RE) metal borohydrides. A new synthesis method provides high yield and high purity products. The trends in crystal structures, thermal behaviour, and magnetic properties are investigated for the entire series of  $\text{RE}(\text{BH}_4)_x$ .
- $\text{NH}_4\text{-M-BH}_4$ : Extensive systematic synthetic work revealed twenty new compounds. The crystal structures are investigated in detail combining synchrotron powder X-ray diffraction and density functional theory, which reveal a variety of compositions and structures ranging from crystalline, solid solutions to polymeric and amorphous compounds. Trends in crystal structures, dihydrogen bonding, thermal- and magnetic properties are presented. The manuscript are in preparation.
- $\text{KB}_3\text{H}_8$ : A manuscript has been submitted based on a study of the structural, hydrogen storage, and ion conductivity properties of  $\text{KB}_3\text{H}_8$ . Three distinct polymorphic transitions are identified for  $\text{KB}_3\text{H}_8$  from a monoclinic ( $\alpha$ ) to a monoclinic ( $\alpha'$ ) at 15 °C via a second-order phase transition and eventually to a cubic ( $\beta$ ) structure at 30 °C by a first-order phase transition. The  $\beta$ -polymorph of  $\text{KB}_3\text{H}_8$  displays high disorder of the  $[\text{B}_3\text{H}_8]^-$  anion and exhibits high  $\text{K}^+$  conductivity of  $\sim 10^{-7} \text{ S}\cdot\text{cm}^{-1}$  above 100 °C.

**Gustav Ek:**

Gustav will also have an extended stay at IFE in Norway together with Jakob where he will investigate the hydrogen storage properties in a number of selected High Entropy Alloys (HEAs). During this time, new projects within the High Entropy Hydrides will be started together with Jakob and Magnus focusing on the electrochemical performance and the synthesis on novel lighter weight materials. During the meeting some of the work Gustav has been conducting on hydrides of High Entropy Alloys and Zintl phases were discussed:

- Gustav and Martin have supervised the first Master student associated with FunHy, Yajie Zhao, who investigated the hydrogen sorption properties within Sc and Y based HEAs. During the work, a travel grant was granted that allowed her to come visit IFE and the JEEP-II reactor for measurements on the PUS diffractometer.
- A paper has been published titled “Hydrogen sorption in TiZrNbHfTa High Entropy Alloy” in Journal of Alloys and Compounds in collaboration with Claudia Zlotea at CNRS in Paris. The paper contained in-situ Synchrotron X-Ray powder diffraction collected at a FunHy beamtime at PETRA III, DESY.
- A new series a ternary quaternary and quinary HEAs have been synthesized and are to be evaluated during the extended stay at IFE.
- The Zintl-phase  $\text{Eu}_3\text{Si}_4$  has been measured in-situ during hydrogenation by Synchrotron X-Ray powder diffraction at the I11 beamline at Diamond Light Source. The results show some interesting behaviour by contraction of one unit cell axis at elevated temperatures. A manuscript is being finalized together with Ulrich Häussermann.
- Another Zintl-phase, NdGa, is currently under consideration for Neutron spectroscopy measurements, but synthesis is proving difficult.

**Magnus M. Nygård:**

It was briefly mentioned that novel mixed-anion, mixed-cation borohydrides  $\text{Na}_2\text{La}_2(\text{BH}_4)_5\text{X}_3$ ,  $X = \text{Cl}, \text{Br}, \text{I}$  has been discovered. The structure was solved in monoclinic space group  $C2/m$  (no. 12) from synchrotron-radiation X-ray powder diffraction (SR-PXD). However, as most of the X-ray scattering originates from La and I, a fully enriched sample utilizing  $^{11}\text{B}$  and D is needed to observe the lighter elements present in the compound by neutron diffraction. Synthesis of such a compound is ongoing. This project has been the basis for the work of a master student, **Emma Berge**. She is currently working with the  $\text{NaBH}_4:\text{REI}_3$ ,  $\text{RE} = \text{Ce}, \text{Pr}, \text{Nd}, \text{Gd}, \text{Tb}, \text{Er}, \text{Y}$  system. In summary, the samples with  $\text{RE} = \text{Ce}, \text{Pr}, \text{Nd}$  form structures that are isostructural to that of  $\text{Na}_2\text{La}_2(\text{BH}_4)_5\text{I}_3$ , while  $\text{RE} = \text{Er}, \text{Y}$  form the well known  $\alpha\text{-RE}(\text{BH}_4)_3$  structure.

However, most of Magnus’ work has been on high-entropy alloys (HEAs) related to the ternary system TiVNb. Thus, most of the presentation was oriented around this. The work has resulted in a total of 18 compounds that all form body-centred cubic (bcc) solid solutions. The synthesis and hydrogen storage properties of these compounds were discussed in detail during the presentation. The fundamental insights can be summarized as:

- Increased concentration of Zr in the HEA alloy induces a hydrogen-induced phase separation that can be traced by *in situ* SR-PXD.
- Large  $[\text{H}]/[\text{M}]$  observed in  $\text{TiVZrNbHf}_{12.5}$  cannot be explained by large local lattice strain alone.
- There is a linear correlation between the valence-electron concentration,  $\text{VEC}$ , and the expansion from the bcc alloy to the face-centred cubic hydride.
- There is also a correlation between the hydrogen desorption onset temperature and the  $\text{VEC}$  of the alloy.
- The desorption kinetics are better in quaternary HEAs as compared to ternary systems.

16.30-17.30 Visit to Neutron reactor Kjeller.  
18- Dinner at the restaurant Fratelli, Lillestrøm (close to hotel)

**Tuesday 18. December**

9.00-9.30 PhD-research project presentation (by Gustav, Magnus and Jakob, 30 min each) and discussions. Planning of new collaborative research activities. (second part)

9.30-10.30 Gustav, Magnus and Jakob continue discussion of their research plans.

Gustav, Magnus and Jakob discuss their plans for secondment together. Development of a final plan for secondment so that local and visiting PhD are together the whole period. The FunHy-PhD's should spend time in other partner laboratories. This needs to be planned with relevant research projects.

Discussion of new research directions.

10:30-11:00 Coffee,

11.00-11.30 Presentation by Martin Sahlberg, overview of activities within NNSP, 'swedness' etc.

Upcoming meetings.

- 23 jan, NNSP steering
- 6-10 may, Swedish Neutron Week, Stockholm
- 1-5 sep, Euromat, Stockholm
- 26-30 aug, JEMS, Uppsala

Also, Martin Sahlberg will take over as Director for SwedNess starting Jan 2019.

Also, lots of funding possibilities from NNSP for exchange and travel between partners. Email to Björn with applications.

Discussion of focus for FunHy, 'where to be visible', and next meeting.

Torben, discussion of next meeting

**Plan for next meetings, 2019:**

21-22. May (lunch-to-lunch): **FunHy and other individual NordForsk meetings**

22-23. May (lunch-to-lunch): **Nordic neutron meeting: Neutrons for sustainable society**

23-24. May (lunch-to-lunch): **Danscatt meeting**

11:30-12:30 Scientific discussions

12.30-13.30 Optional Lunch and Goodbye